



# ***STIC Search Report***

## ***Biotech-Chem Library***

**STIC Database Tracking Number: 129490**

**TO: Shailendra Kumar**  
**Location: 5c03 / 5c18**  
**Tuesday, August 17, 2004**  
**Art Unit: 1621**  
**Phone: 272-0640**  
**Serial Number: 09 / 982105**

**From: Jan Delaval**  
**Location: Biotech-Chem Library**  
**Rem 1A51**  
**Phone: 272-2504**

**[jan.delaval@uspto.gov](mailto:jan.delaval@uspto.gov)**

### **Search Notes**

## SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: S. Kumar Examiner #: 64594 Date: 8/10/04  
 Art Unit: 1621 Phone Number 272-0640 Serial Number: 09/982105  
 Mail Box and Bldg/Room Location: REM 5C03 Results Format Preferred (circle): PAPER DISK E-MAIL  
5C18

If more than one search is submitted, please prioritize searches in order of need.

\*\*\*\*\*

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Process for the production of amides  
 Inventors (please provide full names): Jack S. Warren et al.

Earliest Priority Filing Date: 10/31/2000

What is claimed is:

1. A process for the production of an amide comprising:
  - (a) reacting  $R_1$ -CX and oxygen to form  $R_1$ -COOH, wherein the
    - 5 reacting occurs in the liquid or vapor phase and in the presence of a first catalyst, wherein X is a group that leaves upon oxidation, and wherein  $R_1$  is phenyl, which is unsubstituted or substituted by one or more identical or different radicals selected from  $(C_1-C_{12})$ -alkyl,  $(C_1-C_{12})$ -alkoxy,  $(C_1-C_{12})$ -alkanoyloxy,  $(C_1-C_{12})$ -alkanoyl, amino, hydroxyl,  $-CH_2-O-(C_1-C_{12})$ -alkyl, -NH-
      - 10  $(C_1-C_{12})$ -alkyl, -NH-CO- $(C_1-C_{12})$ -alkyl, or -S- $(C_1-C_{12})$ -alkyl,
    - (b) separating the  $R_1$ -COOH from the mixture formed in step (a), wherein the  $R_1$ -COOH is maintained in a liquid or vapor phase, and
    - (c) reacting the  $R_1$ -COOH maintained in the liquid or vapor phase from step (b) with an amine to form an amide, wherein the reacting occurs in
      - 15 the vapor phase and in the presence of a second catalyst.
  2. The method of claim 1, wherein X is  $-H_3$ .
  3. The method of claim 1, wherein  $R_1$  is meta-methylphenyl.
  4. The method of claim 1, wherein the amine is a secondary amine.
  5. The method of claim 1, wherein the amine is diethylamine.
  6. The method of claim 1, wherein the amine is ethylhexylamine.
  7. The method of claim 1, wherein at least one of the first and second catalysts is a liquid catalyst.

09082105-101701

=> d'his

(FILE 'HOME' ENTERED AT 08:56:40 ON 17 AUG 2004)  
SET COST OFF

FILE 'CASREACT' ENTERED AT 08:57:07 ON 17 AUG 2004

L1 STR  
L2 1 S L1 SAM

FILE 'HCAPLUS' ENTERED AT 08:59:46 ON 17 AUG 2004

L3 1 S WO2001-US49477/AP,PRN  
SEL RN

FILE 'REGISTRY' ENTERED AT 09:02:33 ON 17 AUG 2004

L4 14 S E1-E14  
L5 1 S 108-38-3  
L6 1 S 99-04-7  
L7 1 S 109-89-7  
L8 1 S 134-62-3

FILE 'CASREACT' ENTERED AT 09:03:43 ON 17 AUG 2004

L9 STR L1  
L10 0 S L9  
L11 STR L9  
L12 0 S L11

FILE 'REGISTRY' ENTERED AT 09:05:44 ON 17 AUG 2004

L13 10 S L4 NOT L5-L8  
L14 1 S OXYGEN/CN

FILE 'HCAPLUS' ENTERED AT 09:06:31 ON 17 AUG 2004

FILE 'CASREACT' ENTERED AT 09:08:14 ON 17 AUG 2004

E AMIDE/CT  
L15 2126 S E11  
L16 69325 S AMIDE#/FG.PRO  
L17 69702 S L15,L16  
L18 STR L11  
L19 50 S L18 SAM SUB=L17  
L20 STR L18  
E AMINE/CT  
L21 1515 S E16 AND L17  
L22 63 S E14,E17,E18 AND L17  
L23 49674 S AMINE#/FG.RCT AND L17  
L24 49775 S L21-L23  
L25 25 S L20 SAM SUB=L24  
L26 3777 S L20 FUL SUB=L24  
SAV TEMP L26 KUMAR982/A  
L27 41 S L26 AND OXIDAT?/CW  
L28 35 S L14 AND L26  
L29 59 S L26 AND OXYGEN  
L30 5 S L26 AND O2  
L31 128 S L27-L30  
L32 0 S L31 AND L13  
L33 1 S L31 AND L5  
L34 1 S L31 AND L6  
L35 6 S L31 AND L7  
L36 0 S L31 AND L8  
L37 8 S L33-L35

FILE 'CASREACT' ENTERED AT 09:17:58 ON 17 AUG 2004

L38 0 S (L33 OR L34) AND L35  
L39 0 S L33 AND L34

L40 STR  
L41 42 S L40 SAM SUB=L26  
L42 851 S L40 FUL SUB=L26  
SAV TEMP L42 KUMAR982A/A  
L43 27 S L42 AND L31  
L44 17 S L43 AND (PY<=2000 OR PRY<=2000 OR AY<=2000)

FILE 'REGISTRY' ENTERED AT 09:20:14 ON 17 AUG 2004

FILE 'HCAPLUS' ENTERED AT 09:20:25 ON 17 AUG 2004

L45 15709 S L5  
L46 744 S 1 3 DIMETHYLBENZENE  
L47 476 S BENZENE (S) 1 3 DIMETHYL  
L48 150 S 1 3 XYLENE  
L49 13883 S (M OR META) ( ) XYLENE  
L50 54 S (M OR META) ( ) DIMETHYLBENZENE  
L51 50 S 1 3 ( ) (DIMETHYL OR DI METHYL) ( ) BENZENE  
L52 20960 S L45-L51  
L53 1549 S L6  
L54 1488 S (M OR META) ( ) TOLUIC ACID  
L55 731 S 3 METHYLBENZOIC ACID  
L56 85 S M METHYLBENZOIC ACID  
L57 2868 S L53-L56  
L58 16092 S L7  
L59 630 S ETHYLHEXYLAMINE

FILE 'REGISTRY' ENTERED AT 10:44:08 ON 17 AUG 2004

L60 1 S 104-75-6  
L61 203 S C8H19N/MF  
L62 14 S L61 AND HEXYL  
L63 1 S L61 AND NR>=1  
L64 202 S L61 NOT L63  
L65 200 S L64 AND AMINE  
L66 2 S L64 NOT L65  
L67 47 S L65 AND ((D OR T)/ELS OR 11C# OR 13C# OR 14C# OR C11# OR C13#  
L68 153 S L65 NOT L67  
L69 143 S L68 NOT ION  
L70 142 S L69 NOT LABELED  
L71 142 S L60,L62,L70

FILE 'HCAPLUS' ENTERED AT 10:47:35 ON 17 AUG 2004

L72 11837 S L71  
L73 17175 S DIETHYLAMINE  
L74 32970 S L58,L59,L72,L73  
L75 8 S L52 AND L57 AND L74  
L76 1129 S L8  
L77 476 S DIETHYL ( ) (M OR META) ( ) TOLUAMIDE  
L78 41 S N N DIETHYL 3 METHYLBENZAMIDE  
L79 607 S DEET  
L80 4 S N N DI ETHYL ( ) (M OR META OR 3) ( ) TOLUAMIDE  
L81 99 S N N DIETHYLTOLUAMIDE  
L82 458 S N N DIETHYL ( ) (M OR META OR 3) ( ) TOLUAMIDE  
L83 4 S N N DI ETHYL ( ) (M OR META OR 3) ( ) TOLUAMIDE  
L84 1380 S L76-L83  
L85 2 S L75 AND L84  
L86 4 S L52 AND L57 AND L84  
L87 4 S L85,L86  
L88 3 S L87 AND (L14 OR O2 OR OXYGEN OR OXIDAT?)  
L89 4 S L52 AND L57 AND ET2NH  
L90 3 S L89 AND L84  
L91 4 S L87,L88,L90  
E WARREN J/AU  
E WARREN JACK/AU

L92 13 S E3,E7  
     E WARREN J/AU  
 L93 69 S E3,E20  
     E WESTPHAL D/AU  
 L94 57 S E3,E8  
     E ZOUBECK S/AU  
     E ZOUBEK S/AU  
 L95 3 S E4,E5  
     E EAGLEVIEW/PA,CS  
 L96 26 S E3-E13  
 L97 1 S L92-L96 AND L84  
 L98 1 S L92-L96 AND L52 AND L57  
 L99 4 S L91,L97,L98  
     E WESTPHAL D/AU  
 L100 62 S E3,E4,E6,E8  
 L101 1 S L100 AND L52 AND L57  
 L102 4 S L99,L101

=> fil hcaplus

FILE 'HCAPLUS' ENTERED AT 10:57:29 ON 17 AUG 2004

USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.

PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

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FILE COVERS 1907 - 17 Aug 2004 VOL 141 ISS 8

FILE LAST UPDATED: 16 Aug 2004 (20040816/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d l102 all hitstr tot

L102 ANSWER 1 OF 4 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2002:353423 HCAPLUS

DN 136:371457

ED Entered STN: 12 May 2002

TI Process for the production of amides comprising catalytic oxidation and amidation

IN Warren, Jack; Westphal, David; Zoubek, Steve

PA Eagleview Technologies, Inc., USA

SO PCT Int. Appl., 25 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM C07D

CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)

Section cross-reference(s): 48

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002036559	A2	20020510	WO 2001-US49477	20011023

WO 2002036559 A3 20020906

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,  
 CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,  
 GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,  
 LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PH, PL,  
 PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG,  
 UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM  
 RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY,  
 DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,  
 BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

AU 2002034066

A5

20020515

AU 2002-34066

20011023

PRAI US 2000-244693P

P

20001031

WO 2001-US49477

W

20011023

## CLASS

PATENT NO. CLASS PATENT FAMILY CLASSIFICATION CODES

WO 2002036559 ICM C07D

- AB A process for the production of an amide comprises: (a) reacting R1-CX and **oxygen** to form R1-COOH, wherein the reacting occurs in the liquid or vapor phase and in the presence of a first catalyst, wherein X is a group that leaves upon **oxidation**, and wherein R1 is Ph, which is unsubstituted or substituted by one or more identical or different radicals selected from (C1-C12)-alkyl, (C1-C12)-alkoxy, (C1-C12)-alkanoyloxy, (C1-C12)-alkanoyl, amino, hydroxyl, -CH2-O-(C1-C12)-alkyl, -NH-(C1-C12)-alkyl, -NH-CO-(C1-C12)-alkyl, or -S-(C1-C12)-alkyl; (b) separating the R1-COOH from the mixture formed in step (a), wherein the R1-COOH maintained in the liquid or vapor phase from step (b) with an amine to form an amide, wherein the reacting occurs in the vapor phase and in the presence of a second catalyst. Thus, **N**, **N-di(ethyl)-m-toluamide** was prepared in tube reactors by (1) oxidizing **m-xylene** with **oxygen** in presence of a first catalyst to form **m-toluic acid**, and (2) amidizing the acid with **diethylamine** in presence of a second catalyst. The first and second catalysts are selected from one or more of MgO, TiO2, ZrO2, ZnO, CeO2, Ce2O3, tungsten heteropolyacid, hydroxyapatite, cobalt octoate, and copper octoate.
- ST **diethyl meta toluamide** prodn catalytic  
**oxidn** amidation; xylene **oxidn** **diethylamine**  
 amidation process
- IT Heteropoly acids  
 RL: CAT (Catalyst use); USES (Uses)  
 (Tungsten catalyst; in production of amides comprising catalytic **oxidation** and amidation)
- IT Amidation catalysts  
**Oxidation** catalysts  
 (in production of amides comprising catalytic **oxidation** and amidation)
- IT Amidation  
**Oxidation**  
 (production of amides comprising catalytic **oxidation** and amidation)
- IT Amides, preparation  
 RL: IMF (Industrial manufacture); PREP (Preparation)  
 (production of amides comprising catalytic **oxidation** and amidation)
- IT Amines, reactions  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (secondary; in production of amides comprising catalytic **oxidation** and amidation)
- IT Reactors  
 (tubular; in production of amides comprising catalytic **oxidation** and amidation)
- IT 136-52-7, Cobalt octoate 1306-06-5, Hydroxyapatite 1306-38-3, Cerium oxide (CeO2), uses 1309-48-4, Magnesium oxide (MgO), uses 1314-13-2,

Zinc oxide (ZnO), uses 1314-23-4, Zirconium oxide (ZrO<sub>2</sub>), uses 1345-13-7, Cerium oxide (Ce<sub>2</sub>O<sub>3</sub>) 7440-33-7D, Tungsten, Heteropoly acid derivative 13463-67-7, Titanium oxide (TiO<sub>2</sub>), uses 20543-04-8, Copper octoate

RL: CAT (Catalyst use); USES (Uses)

(catalyst; in production of amides comprising catalytic oxidation and amidation)

IT 108-38-3, **m-Xylene**, reactions 109-89-7

, **Diethylamine**, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)

(in production of **N,N-di(ethyl)-**

**m-toluamide** comprising catalytic oxidation and amidation)

IT 99-04-7P, **m-Toluic acid**

RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)

(intermediate; in production of **N,N-di(**

**ethyl)-m-toluamide** comprising catalytic oxidation and amidation)

IT 134-62-3P, **N,N-Di(ethyl)-**

**m-toluamide**

RL: IMF (Industrial manufacture); PREP (Preparation)

(production of **N,N-di(ethyl)-**

**m-toluamide** comprising catalytic oxidation and amidation)

IT 108-38-3, **m-Xylene**, reactions 109-89-7

, **Diethylamine**, reactions

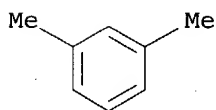
RL: RCT (Reactant); RACT (Reactant or reagent)

(in production of **N,N-di(ethyl)-**

**m-toluamide** comprising catalytic oxidation and amidation)

RN 108-38-3 HCAPLUS

CN Benzene, 1,3-dimethyl- (9CI) (CA INDEX NAME)



RN 109-89-7 HCAPLUS

CN Ethanamine, N-ethyl- (9CI) (CA INDEX NAME)



IT 99-04-7P, **m-Toluic acid**

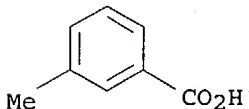
RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)

(intermediate; in production of **N,N-di(**

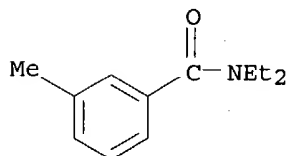
**ethyl)-m-toluamide** comprising catalytic oxidation and amidation)

RN 99-04-7 HCAPLUS

CN Benzoic acid, 3-methyl- (9CI) (CA INDEX NAME)



IT 134-62-3P, N,N-Di(ethyl)-  
**m-toluamide**  
 RL: IMF (Industrial manufacture); PREP (Preparation)  
 (production of N,N-di(ethyl)-  
**m-toluamide** comprising catalytic oxidation and  
 amidation)  
 RN 134-62-3 HCAPLUS  
 CN Benzamide, N,N-diethyl-3-methyl- (9CI) (CA INDEX NAME)



L102 ANSWER 2 OF 4 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1997:51199 HCAPLUS

DN 126:74598

ED Entered STN: 24 Jan 1997

TI Process for preparing N,N-diethyl-  
**meta-toluamide**

IN Shejn, Samuil M.; Makshanova, Nina P.; Pomogaeva, Lyudmila S.;  
 Baranetskaya, Galina T.; Tulupov, Nikolaj S.; Balabanov, Valerij Yu.;  
 Tarasova, Nelli V.

PA Moskovskoe Nauchno-Proizvodstvennoe Ob"edinenie "niopik", Russia  
 SO Russ.

From: Izobreteniya 1996, (9), 217.

CODEN: RUXXE7

DT Patent

LA Russian

IC ICM C07C233-65

ICS C07C231-02

CC 25-19 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds)  
 Section cross-reference(s): 5

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	RU 2057118	C1	19960327	RU 1992-12633	19921215
PRAI	RU 1992-12633		19921215		

CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
RU 2057118	ICM	C07C233-65
	ICS	C07C231-02

AB The title compound (i.e., **DEET**) is prepared by amidation of  
**m-toluic acid** with **Et2NH** at  
 200-220°, with azeotropic distillation of formed H2O of reaction, using a  
 complex-forming trivalent P or B compound as catalyst, and an aromatic  
 hydrocarbon solvent (benzene, toluene, or **m-xylene**).

ST diethylmetatoluamide; toluamide diethyl meta; **DEET**; amidation  
 toluic acid **diethylamine**

IT Amidation

Amidation catalysts

Insect repellents

(preparation of N,N-diethyl-**meta-**  
**toluamide** by amidation of toluic acid with **diethylamine**  
 )

IT 7440-42-8D, Boron, trivalent compds., uses 7723-14-0D, Phosphorus,

trivalent compds., uses

RL: CAT (Catalyst use); USES (Uses)  
(catalyst; preparation of **N,N-diethyl-meta-toluamide** by amidation of toluic acid with **diethylamine**)

IT 134-62-3P, **N,N-Diethyl-m-toluamide**

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)  
(preparation of **N,N-diethyl-meta-toluamide** by amidation of toluic acid with **diethylamine**)

IT 99-04-7, **m-Toluic acid**

109-89-7, **Diethylamine**, reactions

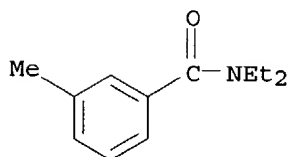
RL: RCT (Reactant); RACT (Reactant or reagent)  
(preparation of **N,N-diethyl-meta-toluamide** by amidation of toluic acid with **diethylamine**)

IT 134-62-3P, **N,N-Diethyl-m-toluamide**

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)  
(preparation of **N,N-diethyl-meta-toluamide** by amidation of toluic acid with **diethylamine**)

RN 134-62-3 HCAPLUS

CN Benzamide, N,N-diethyl-3-methyl- (9CI) (CA INDEX NAME)



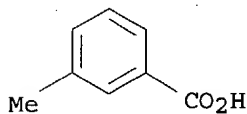
IT 99-04-7, **m-Toluic acid**

109-89-7, **Diethylamine**, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)  
(preparation of **N,N-diethyl-meta-toluamide** by amidation of toluic acid with **diethylamine**)

RN 99-04-7 HCAPLUS

CN Benzoic acid, 3-methyl- (9CI) (CA INDEX NAME)



RN 109-89-7 HCAPLUS

CN Ethanamine, N-ethyl- (9CI) (CA INDEX NAME)



DN 115:135699  
 ED Entered STN: 05 Oct 1991  
 TI Process for the preparation of **N,N-diethyltoluamides** by **oxidation**, chlorination, and amidation of xylenes  
 IN Koch, Joachim; Rudnick, Klaus; Staschok, Axel; Till, Lothar  
 PA Berlin-Chemie A.-G., Germany  
 SO Ger. (East), 3 pp.  
 CODEN: GEXXA8  
 DT Patent  
 LA German  
 IC ICM C07C103-76  
 CC 25-19 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds)  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DD 288823	A5	19910411	DD 1989-332790	19890919
PRAI	DD 1989-332790		19890919		

## CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
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DD 288823	ICM	C07C103-76
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OS CASREACT 115:135699

AB A process for the preparation of **N,N-diethyltoluamides** from xylene-enriched C8-aroms. comprises the **oxidation**, carbonyl chloride formation and, diethylamidation of said C8-aroms. A mixture of C8-aroms., 0.1-0.6% 99% AcOH and 0.01-0.3% 30% aqueous manganese bromide is heated to 180-200°, the aqueous condensate is removed, pressure is reduced to 0.1 MPa and the condensate is treated with SOCl<sub>2</sub> at 100-120°; the acidic fraction is removed and **Et<sub>2</sub>NH** and H<sub>2</sub>O are added to the reactor. Remaining C8-aroms. are recycled and **N,N-diethyltoluamide** is fractionated at 1.8-2.2 kPa at 150-165° head temperature. A Ti reactor was charged with 10.2 kg C8-aroms. (70% by weight **m-xylene**, 10% by weight o-xylene, 20% by weight p-xylene), 59 g 30% aqueous manganese bromide, and 50 g 99% AcOH

at

190° and 2.0 MPa and air was added to give a mixture containing 62% toluic acids, 38% xylenes, 800 ppm H<sub>2</sub>O and 90 ppm byproducts. The oxidized mixture was treated with 9.1 kg SOCl<sub>2</sub> for 2 h at 110-120°. The resulting acid chloride mixture was cooled to 30° and treated with 8.1 kg **Et<sub>2</sub>NH** and 11.5 L H<sub>2</sub>O; the yield of **N,N-diethyltoluamide** was 65%.

ST toluamide; xylene **oxidn** chlorination amidation

IT Aromatic hydrocarbons, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)

(C8, xylene-rich, **oxidation**, chlorination, and sequential diethylamination of)

IT 1330-20-7, Xylene, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)

(oxidation, chlorination, and sequential diethylamidation of, diethyltoluamide from)

IT 95-47-6, o-Xylene, reactions 106-42-3, p-Xylene, reactions

108-38-3, **m-Xylene**, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)

(oxidation, chlorination, and sequential diethylamination of, toluamide from)

IT 134-62-3P, **DEET** 2728-04-3P, N,N-Diethyl-o-toluamide2728-05-4P, N,N-Diethyl-P-toluamide 26545-51-7P, **N,N****-Diethyltoluamide**

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

IT 99-04-7P, **m-Toluic acid** 99-94-5P

118-90-1P, o-Toluic acid

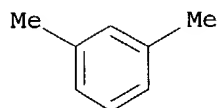
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation, chlorination, and sequential diethylamidation of)

IT 108-38-3, m-Xylene, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)  
(oxidation, chlorination, and sequential diethylation of, toluamide from)

RN 108-38-3 HCAPLUS

CN Benzene, 1,3-dimethyl- (9CI) (CA INDEX NAME)

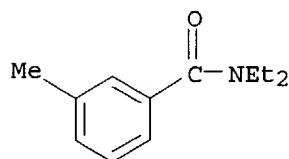


IT 134-62-3P, DEET

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 134-62-3 HCAPLUS

CN Benzamide, N,N-diethyl-3-methyl- (9CI) (CA INDEX NAME)

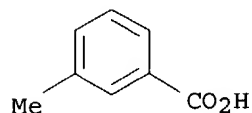


IT 99-04-7P, m-Toluic acid

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation, chlorination, and sequential diethylamidation of)

RN 99-04-7 HCAPLUS

CN Benzoic acid, 3-methyl- (9CI) (CA INDEX NAME)



L102 ANSWER 4 OF 4 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1970:487657 HCAPLUS

DN 73:87657

ED Entered STN: 12 May 1984

TI N,N-Diethyltoluamides

IN Schulze, Werner; Thiele, Martin

SO Ger. (East), 2 pp.

CODEN: GEXXA8

DT Patent

LA German

IC C07C

CC 25 (Noncondensed Aromatic Compounds)

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DD 71761		19700320	DD	19681101

## CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
DD 71761	IC	C07C

AB A one-step procedure involving the **oxidation** of **m-xylene** followed by conversion of the resulting **m-toluic acid** to the N,N-diethylamide via the acid chloride was described. A stream of air was passed at 0.5 l./min through 80 ml boiling **m-xylene** containing 0.5 g cobalt oleate and water removed. After 6 hr, titration indicated 12 g **m-toluic acid** had formed. The **oxidation** mixture was treated with 17 ml SOCl<sub>2</sub> and refluxed 2 hr, excess SOCl<sub>2</sub> removed, and the acid chloride treated with a solution of 19 g Et<sub>2</sub>NH in 30 ml xylene at low temperature to yield 15 g title compound, b<sub>0.15</sub> 155-60°.

ST toluamides diethyl

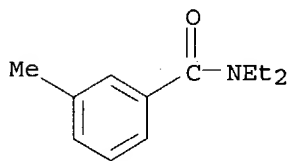
IT 1330-20-7  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(**oxidation** of, toluic acid derivs. by)

IT 134-62-3P 2728-04-3P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

IT 134-62-3P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 134-62-3 HCAPLUS

CN Benzamide, N,N-diethyl-3-methyl- (9CI) (CA INDEX NAME)



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